

Appln No. 10/767,875
Amdt date September 7, 2007
Reply to Office action of June 7, 2007

REMARKS/ARGUMENTS

In the Office action dated June 7, 2007, the examiner indicated that the Information Disclosure Statement submitted on January 16, 2007 did not include copies of the cited reference. Applicant re-submitted this Information Disclosure Statement along with a copy of the cited foreign reference on June 18, 2007. A copy of this re-submission, including the foreign reference appears in the image file wrapper on the Office's PAIR page for this application. However, for convenience, applicant attaches to this response a copy of the January 16, 2007 Information Disclosure Statement and a copy of the cited foreign reference.

Turning to the claims, the examiner maintained the objection to claim 12 under 37 CFR §1.75 and MPEP §706.03(k) as being a substantial duplicate of claim 10. In maintaining this objection, the examiner argues that a positive electrode and a separator are "inherent to rechargeable lithium batter[ies]," and therefore that claim 12 is a substantial duplicate of claim 10. However, applicant has amended claim 10 to recite a negative electrode for a rechargeable lithium battery. MPEP 706.03(k) notes that a "mere difference in scope between claims" is enough to avoid an objection under that section. As claim 10 is now directed to a negative electrode, and claim 12 is directed to a lithium rechargeable battery, applicant submits that the claims are sufficiently different to obviate the examiner's objection under CFR §1.75 and MPEP §706.03(k).

In addition, the examiner maintained the rejection of claims 10 and 12 under 35 U.S.C. §102(b) as allegedly anticipated by, or in the alternative under 35 U.S.C. §103(a) as allegedly obvious over Idota (U.S. Patent No. 5,618,640). In maintaining this rejection, the examiner simply states that "Idota discloses a rechargeable lithium battery with a negative electrode consisting essentially of a carbonaceous material as the negative electrode active material." Office action, page 4. However, Idota fails to teach a negative electrode *consisting essentially of*

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a carbonaceous material. In fact, Idota expressly teaches away from a negative electrode consisting essentially of a carbonaceous material. At Column 1, lines 17-32, Idota notes that metallic lithium precipitates on carbonaceous negative active materials upon overcharge or rapid charge. In addition, Idota notes that carbonaceous materials have low density and low capacity per unit volume, thereby limiting the discharge capacity of the battery. In response to these limitations, Idota discloses the use of a $M^1M^2_pM^4_q$ compound as the main component of the negative active material.

Moreover, the transitional phrase "consisting essentially of," used in claims 10 and 12, excludes those materials that would materially affect the basic and novel characteristics of the claimed invention. Idota discloses a negative active material in which the main component is a $M^1M^2_pM^4_q$ compound in which M^1 and M^2 may be Si, Ge, Sn, Pb, P, B, Al, As or Sb and M^4 may be O, S, Se or Te. See Column 4, lines 19-33. The addition of a $M^1M^2_pM^4_q$ compound to a carbonaceous negative active material, as claimed in the present application, would materially affect the characteristics of the claimed invention, as evidenced by the disclosure in Idota. Therefore, Idota fails to teach or suggest a negative electrode *consisting essentially of* a carbonaceous material, and claims 10 and 12 are allowable over Idota.

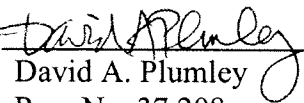
The examiner also asserted that claims 10 and 12 are product-by-process claims by virtue of the limitations directed to the amount of gas generated during initial charging and discharging. While applicant disagrees with this characterization, applicant also notes that the products, namely, the negative active material and the rechargeable lithium battery, are allowable over Idota independent of this limitation, as discussed above.

Claims 10 and 12 remain pending in this application. By this amendment, applicant has amended claim 10. The amendments find full support in the original specification, claims and drawings. No new matter is presented. In light of the above amendments and remarks, applicant submits that both of claims 10 and 12 are in condition for allowance. Applicant therefore

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respectfully requests reconsideration and a timely indication of allowance. However, if there are any remaining issues that can be addressed by telephone, applicant invites the examiner to contact applicant's counsel at the number indicated below.

Respectfully submitted,
CHRISTIE, PARKER & HALE, LLP

By 
David A. Plumley
Reg. No. 37,208
626/795-9900


LES/les

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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

I hereby certify that this correspondence is being electronically filed with the United States Patent and Trademark Office on January 16, 2007.


Diane L. Goss

Applicant : Young-Jun Kim, et al. Confirmation No. 4124
Application No. : 10/767,875
Filed : January 29, 2004
Title : METHOD OF PREPARING NEGATIVE ELECTRODE FOR
RECHARGEABLE LITHIUM BATTERY, METHOD OF FABRICATING
RECHARGEABLE LITHIUM BATTERY AND RECHARGEABLE
LITHIUM BATTERY
Grp./Div. : 1745
Examiner : Keith D. Walker
Docket No. : 51813/Y35

INFORMATION DISCLOSURE STATEMENT WITH FEE
UNDER 37 CFR §§ 1.97(d) AND 1.17(p)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Post Office Box 7068
Pasadena, CA 91109-7068
January 16, 2007

Commissioner:

In compliance with the duty of disclosure under 37 CFR §§ 1.56, 1.97 and 1.98, and in accordance with the provisions in the Manual of Patent Examining Procedure §§ 609 and 707.05(b), enclosed is FORM PTO/SB/08A/B listing a reference that is known to applicant. A copy of the listed reference is enclosed.

It is respectfully requested that the listed reference be considered in the examination of this application and identified on the list of references cited on the patent issuing for this application. Applicant also requests that an initialed copy of FORM PTO/SB/08A/B be entered in the application file and returned to applicant with the next communication from the Office in accordance with MPEP § 609.

Application No. 10/767,875

Applicant's undersigned attorney hereby certifies, in accordance with 37 CFR § 1.97(e)(1), that each item of information contained in the information disclosure statement was first cited in a communication from any foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement.

Enclosed, via deposit account, is the processing fee of \$180 as required by 37 CFR § 1.17(p). The Commissioner is hereby authorized to charge any fees which may be required by this paper to Deposit Account No. 03-1728. Please show our docket number with any Deposit Account transaction.

Respectfully submitted,

CHRISTIE, PARKER & HALE, LLP

By 

D. Bruce Prout

Reg. No. 20,958

626/795-9900

DBP/dg

Enclosures: Copy of IDS

Form PTO/SB/08A/B, w/reference

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FORM PTO/SB/08A/B (10-01) Substitute for PTO-1449A/B INFORMATION DISCLOSURE STATEMENT BY APPLICANT (use as many sheets as necessary)	Attorney Docket Number	51813/Y35
	Application Number	10/767,875
	Filing Date	January 29, 2004
	Applicant(s)	Young-Jun Kim, et al.
	Group Art Unit	1745
	Examiner Name	Keith D. Walker

U.S. PATENT DOCUMENTS				
EXAMINER INITIALS	Cite No. ¹	DOCUMENT NUMBER Number - Kind Code ² (If Known)	PUBLICATION DATE MM-DD-YYYY	NAME OF PATENTEE

FOREIGN PATENT DOCUMENTS					
EXAMINER INITIALS	Cite No. ¹	Foreign Patent Document Country Code ² - Number ³ - Kind Code ⁴ (If Known)	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	T ⁵ (✓)
		CN 1212473A	03-31-1999	Ma et al.	English Abstract

OTHER DOCUMENTS		
EXAMINER INITIALS	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article, title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.

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EXAMINER SIGNATURE		DATE CONSIDERED	
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609; Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. ¹Applicant's unique citation designation number (optional). ²See Kinds Codes of USPTO Patent Documents at www.pto.gov or MPEP 901.4. ³Enter Office that issued the document, by the two-letter code (WIPO standard ST.3). ⁴For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁶Applicant is to place a check mark here if English Language Translation is attached.

Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

DBP/dg

Acknowledgement Receipt

The USPTO has received your submission at **14:04:03** Eastern Time on **16-JAN-2007** by Deposit Account: 031728.

\$ **180** fee paid by e-File via *RAM* with Confirmation Number: 2471.

eFiled Application Information

EFS ID	1440541
Application Number	10767875
Confirmation Number	4124
Title	Method of preparing a negative electrode for a rechargeable lithium battery, method of fabricating a rechargeable lithium battery, and a rechargeable lithium battery
First Named Inventor	Young-Jun Kim
Customer Number or Correspondence Address	23363
Filed By	D. Bruce Prout/Diane Goss
Attorney Docket Number	51813/DBP/Y35
Filing Date	29-JAN-2004
Receipt Date	16-JAN-2007
Application Type	Utility

Application Details

Submitted Files	Page Count	Document Description	File Size	Warnings
51813ids.pdf	3		84621 bytes	◆ PASS
		Document Description	Page Start	Page End
		Information Disclosure Statement (IDS) Filed	1	2
		Information Disclosure Statement (IDS) Filed	3	3
51813reference.pdf	10	Foreign Reference	528363 bytes	◆ PASS
fee-info.pdf	2	Fee Worksheet (PTO-06)	8229 bytes	◆ PASS

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53 (b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

If you need help:

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- Call the Patent Electronic Business Center at (866) 217-9197 (toll free) or e-mail EBC@uspto.gov for specific questions about Patent e-Filing.
- Send general questions about USPTO programs to the USPTO Contact Center (UCC).
- If you experience technical difficulties or problems with this application, please report them via e-mail to Electronic Business Support or call 1 800-786-9199.

Preparation of lithium ion battery carbon material negative pole

Publication number	CN1212473
Publication date	1999.07.28
Inventor	MA SHUHLA (CHINA) NING XIAOYI (CHINA)
Applicant	CHANGCHUN APPLIED CHEMISTRY CO.
Classification	
Int. Cl. Class.	H01M4/04, H01M4/15, H01M4/16
European patent	
Application number	CN1196701800.0
Priority number(s)	CN99711860.0

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Abstract of CN1212473

The preparation method of carbon material catelectrode of lithium ion battery is characterized by that it adopts the electrolytic salt containing lithium ion or its inorganic or organic solution containing oxygen compound to treat the graphite or intermediately-graphitized carbon material electrode, and on the electrode surface a layer of compact lithium ion conducted solid electrolytic film is preformed so as to raise the first charging and discharging efficiency of graphites or intermediately-graphitized carbon material catelectrode of lithium ion battery and stabilize its circulation volume. The electrolytic film prepared by said invented method can greatly improve the properties of graphite electrode, and also possesses the obvious action for improving random layer structure carbon.

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[19]中华人民共和国国家知识产权局

[51]Int. Cl⁶

H01M 4/04

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[43]公开日 1999年3月31日

[11]公开号 CN 1212473A

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代理人 曹桂珍

权利要求书1页 说明书7页 附图页数0页

[54]发明名称 锂离子电池碳材料负极的制备

[57]摘要

本发明属于锂离子电池碳材料负极的制备方法。

本发明采用含锂离子电解质盐或其含氧化合物的无机或有机溶液处理石墨或中等石墨化程度的碳材料电极,在电极表面预生成一层致密的锂离子导通的固体电解质薄膜以提高锂离子电池石墨类或中等石墨化程度碳材料负极的首次充放电效率及稳定循环容量。

本发明方法制备的电解质薄膜对石墨类电极性能改善幅度较大、对乱层结构碳也有明显的改善作用。

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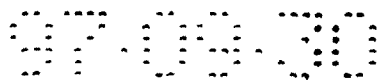
权 利 要 求 书

1. 一种锂离子电池碳材料负极的制备方法, 其特征在于选用的碳材料为石墨、中间相碳微球、碳纤维; 选用的含锂离子电解质盐及其含氧化合物为 Li_2CO_3 、 LiOH 、 Li_2O 、 LiCl , 用于溶解含锂离子电解质盐或其含氧化合物的溶剂为 H_2O 、乙醇、四氢呋喃, 溶液浓度控制在重量百分比浓度 1—20% 之间;

具体制备步骤如下:

1) 将石墨或中等石墨化程度碳粉置于称量瓶中, 按 3% 的重量比加入 5% 的聚四氟乙烯水乳液, 再加入无水乙醇至称量瓶 1/2 处, 放入搅拌子, 在密闭状态下电磁搅拌 12 小时混合均匀后, 自然状态下待乙醇挥发至溶液呈粘稠状后, 用 $60\text{kg}/\text{cm}^2$ 的压力将混合物紧压在经过碱煮除油处理的干净镍网上, 真空加热 160°C 干燥 48 小时, 移入手套箱中, 称量, 密闭保存, 得到待进行电解质膜沉积处理的粉末电极;

2) 将经石墨或中等石墨化程度碳粉制得的粉末电极置于培养皿中, 加入含锂离子电解质盐或其含氧化合物的无机或有机溶液, 自然放置 48—96 小时, 取出, 置于滤纸上, 自然放置 48—96 小时后置于真空烘箱中, 真空加热 $100—200^\circ\text{C}$ 、干燥 48 小时以上, 得到预沉积膜电极。



说明书

锂离子电池碳材料负极的制备

本发明属于锂离子电池碳材料负极的制备方法。

新型锂离子电池由于采用嵌锂碳材料替代金属锂作为负极,使其既保留了锂电池高比能量的优点,又解决了循环寿命和安全性差的问题。随着锂离子电池的发展,在本世纪末将形成锂离子电池与氢镍、镉镍电池三分天下的局面,而成为通讯和信息产业的能源支柱产品。

然而,碳材料的负极电池性能极大地受到碳材料本身的结构、所用的电解质溶液的性质的影响。碳材料由六角形的石墨片层网面堆积而成,对于确定的微晶显示出特有的边缘(edge surface)、底面(basal plane)特征,具有各向异性反应性质。其中,作为电极的碳材料的微晶的边缘部分具有较强的电荷密度、电场强度较大,具有较高的反应性;相反,碳材料微晶的底面部分则电荷密度较小、电场较弱,反应性较差。碳上所发生的电化学反应主要集中发生于边缘部位。同时,锂离子电池所用的一些典型的溶剂及电解质盐由于本身各自的内在结构因素,其电化学还原分解反应的动力学及热力学性质也显著不同。比如,最常见的溶剂碳酸丙烯酯(PC)就极易发生还原分解反应,反应速度较快。首次充电过程中,溶剂及电解质盐的不可逆电化学还原分解反应产物锂的烷氧基碳酸盐将实时地沉积在碳电极表面,生成一层电子绝缘、锂离子导通的固体电解质膜,这层钝化膜的致密性质将强烈地影响着碳材料负极的电池性能。一层薄而致密的电极表面钝化膜的生成能够阻止锂离子的溶剂化共嵌入,保证锂离子在碳基底的石墨层间能够可逆地嵌入脱出,使碳电极具有好的锂离子电池负极性能。碳材料微晶的边缘、底面之间在量、尺寸及反应性之间的差别以及电解液组分还原分解的动力学速度决定了所生成的电极表面钝化膜的致密性质。

比如,石墨是一类极具发展前途的锂离子电池用负极碳材料。石墨作为锂离子电池的负极,与其它结构的碳材料相比具有充放电平台低,电压稳定(0.0—0.2V vs. Li^+/Li)的优点。但是由于石墨的石墨化程度较大,碳微晶的边缘、底面之间的差别较大,反应不均匀性较强,电解液的还原分解反应主要发生于微晶的边缘部分,所生成的钝化膜的致密性质较差,在充电过程中,锂离子插入石墨层间时易发生溶剂化锂离子的共嵌入,引起石墨层的膨胀、崩溃等不可逆过程发生,使得石墨材料不适于直接作为二次锂电池的电极材料。特别

是在含还原分解反应动力学速度较快, 极易还原分解的溶剂碳腈丙烯酯(PC)的电解液中表现的更加显著, 电极的首次充放电效率、锂离子的嵌入容量极低, 根本不能使用。其它的非石墨类碳材料也都不同程度地存在着首次充放电效率低的问题, 使得电池密闭体系中锂源即正极材料的用量较大, 电池的生产成本较高, 不利于电池的大规模工业化生产。

一些研究者曾提出在电解液中添加冠醚 [J. O. Besenhard, M. W. Wagner, M. Winter, D. D. Jannakoudakis, P. D. Jannakoudakis and E. Theodoridou, *J. Power Sources*, 43 - 44, (1993)413], 将电池体系置于高压的二氧化碳气氛 [Z. X. Shu, R. S. McMillan and J. J. Murray, *J. Electrochem. Soc.*, 140(4), 1993, 922] 及对碳材料进行预化学还原处理 [马树华, 国汉举, 李季, 梁洪泽, 景遐斌, 王佛松. 电化学, 1996, 2(4): 413] 等, 这些改善办法都能够不同程度地提高碳材料的电极性能, 具有一定的实用效果。

上述改性办法都是着眼于改善由电极体系本身所自发形成的钝化膜的致密性质或者增大溶剂化锂离子的尺寸, 阻止锂离子的溶剂化共嵌入以对碳负极的电极性能进行改性, 这只是解决问题的一个方面。

本发明的目的是采用含锂离子电解质盐或其含氧化合物的无机或有机溶液体系处理石墨或中等石墨化程度碳材料电极, 在电极表面生成一层致密的锂离子导通的固体电解质薄膜以提高锂离子电池石墨类或中等石墨化程度碳材料负极的首次充放电效率及稳定循环容量。

本发明提出的改性办法摒弃了电池体系(电极基底、电解液体系等)本身各自所具有的内在缺陷的影响, 对于锂离子电池碳材料负极的电池性能进行改善。

本发明选用的碳材料为石墨、中间相碳微球、碳纤维; 选用的含锂离子电解质盐及其含氧化合物为 Li_2CO_3 、 LiOH 、 Li_2O 、 LiCl , 用于溶解含锂离子电解质盐或其含氧化合物的溶剂为 H_2O 、乙醇、四氢呋喃, 溶液浓度控制在重量百分比浓度 1—20% 之间;

具体制备步骤如下:

1) 将石墨或中等石墨化程度碳粉置于称量瓶中, 按 3% 的重量比加入 5% 的聚四氟乙烯水乳液, 再加入无水乙醇至称量瓶 1/2 处, 放入搅拌子, 在密闭状态下电磁搅拌 12 小时混合均匀后, 自然状态下待乙醇挥发至溶液呈粘稠状后, 用 60kg/cm^2 的压力将混合物紧压在经过碱煮除油处理的干净镍网上, 真空加热 160°C 干燥 48 小时, 移入手套箱中, 称量, 密闭保存, 得到待进行电解质膜沉积处理的粉末电极;

2) 将经石墨或中等石墨化程度碳粉制得的粉末电极置于培养皿中, 加入含锂离子电解质盐或其含氧化合物的无机或有机溶液, 自然放置 48 — 96 小时, 取出, 置于滤纸上, 自然放置 48 — 96 小时后置于真空烘箱中, 真空加热 100 — 200 °C、干燥 48 小时以上, 得到预沉积膜电极。

本发明对石墨类碳材料电极的电池性能改善幅度较大。经过本发明处理后得到的预沉积膜电极, 能够使用含易还原溶剂碳酸丙烯酯(PC, Propylene Carbonate)的电解液; 同时, 本发明对非石墨类的中等石墨化程度乱层结构碳材料电极也具有明显的改善作用。

经本发明沉积固体电解质膜的石墨或碳粉末电极表面呈暗灰白色, 表面电子电导为 10^{-10} S/cm 以下, 电子绝缘, 但锂离子导通, 可以有效地抑制溶剂化锂离子的共嵌入, 避免石墨层的崩溃, 提高电极的锂离子嵌入脱出充放电容量至 270 — 340mAh/g, 并且可以抑制溶剂的进一步还原分解而提高首次充放电效率至 60 — 91%。

本发明制备工艺简单可行, 易于工业实施。

本发明提供的实施例如下:

实施例 1:

将 1 克中间相碳微球石墨粉 MCMB28 (Mesocarbon microbeads, 2800 °C) 置于称量瓶中, 按 3% 的重量比加入 5% 的聚四氟乙烯水乳液, 再加入无水乙醇至称量瓶 1/2 处, 放入搅拌子, 在密闭状态下电磁搅拌 12 小时混合均匀后, 自然状态下候乙醇挥发至溶液呈粘稠状后, 用 60kg/cm^2 的压力将混合物紧压在经过碱煮等除油处理的干净镍网上, 真空加热 160 °C 干燥 48 小时, 移入手套箱中, 称量, 密闭保存。

将 MCMB28 石墨粉按上述方法制备的粉末电极置于培养皿中, 加入 1% 的 LiOH 水溶液, 使溶液液面超出电极表面 2—3 厘米, 自然放置 96 小时, 取出, 置于滤纸上, 自然放置 96 小时后置于真空烘箱中, 真空加热 150 °C、干燥 48 小时以上。移入手套箱中装配电池进行单电极的电池性能的测试。

典型的电极电池性能如下:

充放电电流密度: 0.5mA/cm^2

(1). 在 1M LiClO₄ EC/DEC 电解液中

首次放电容量: 290.8mAh/g, 充放电效率: 69.5%

第二次放电容量: 287.2mAh/g, 充放电效率: 96.3%

第十次放电容量: 289.1mAh/g, 充放电效率: 98.7%

首次充放电效率提高 20.8%, 稳定循环容量提高 140.2mAh/g

(2).在 1M LiClO₄ PC/DME 电解液中

首次放电容量:291.3mAh/g, 充放电效率:62.5%

第二次放电容量:261.8mAh/g, 充放电效率:94.4%

第十次放电容量:266.7mAh/g, 充放电效率:90.7%

首次充放电效率提高 19.9%, 稳定循环容量提高 185.8mAh/g

实施例 2:

将MCMB28石墨粉按实施例1中所述方法制备的粉末电极置于培养皿中, 加入20%的Li₂CO₃水溶液, 使溶液液面超出电极表面2—3厘米, 自然放置96小时, 取出, 置于滤纸上, 自然放置96小时后置于真空烘箱中, 真空加热150℃、干燥48小时以上, 移入手套箱中装配电池进行单电极的电池性能的测试。

将经过一定次数的充放电循环以后的电极取出在扫描电子显微镜下观察时, 可以观察到表面晶体膜比较均匀、致密、平滑, 高低轮廓清晰, 沉积的晶体膜层随着碳电极基底本身的起伏变化而起伏变化, 与电极基底的附着比较紧密, 基本没有龟裂产生。

典型的电极电池性能如下:

充放电电流密度:0.5mA/cm²

(1).在 1M LiClO₄ EC/DEC 电解液中

首次放电容量:296.1mAh/g, 充放电效率:90.3%

第二次放电容量:290.5mAh/g, 充放电效率:99.8%

第十次放电容量:288.1mAh/g, 充放电效率:97.3%

首次充放电效率提高 41.6%, 稳定循环容量提高 139.2mAh/g

(2).在 1M LiClO₄ PC/DME 电解液中

首次放电容量:273.5mAh/g, 充放电效率:88.7%

第二次放电容量:269.4mAh/g, 充放电效率:97.9%

第十次放电容量:258.6mAh/g, 充放电效率:98.3%

首次充放电效率提高 46.1%, 稳定循环容量提高 177.7mAh/g

实施例 3:

将天然石墨粉(NG)按实施例1中所述方法制备的粉末电极置于培养皿中, 加入5%的LiCl水溶液, 使溶液液面超出电极表面2—3厘米, 自然放置96小时, 取出, 置于滤纸上, 自然放置96小时后置于真空烘箱中, 真空加热150℃、干燥48小时以上, 移入手套箱中装配电池进行单电极的电池性能的测试。

典型的电极电池性能如下:

充放电电流密度:0.5mA/cm²

(1).在 1M LiClO₄ EC/DEC 电解液中

首次放电容量:327.1mAh/g, 充放电效率:84.2%

第二次放电容量:311.6mAh/g, 充放电效率:93.3%

第十次放电容量:307.2mAh/g, 充放电效率:95.7%

首次充放电效率提高 34.4%, 稳定循环容量提高 64.5mAh/g

(2).在 1M LiClO₄ PC/DME 电解液中

首次放电容量:279.3mAh/g, 充放电效率:75.7%

第二次放电容量:240.8mAh/g, 充放电效率:89.6%

第十次放电容量:210.5mAh/g, 充放电效率:94.7%

首次充放电效率提高 59.9%, 稳定循环容量提高 185.1mAh/g

实施例 4:

将天然石墨粉(NG)按实施例1中所述方法制备的粉末电极置于培养皿中, 加入5%的LiCl四氢呋喃溶液, 使溶液液面超出电极表面2—3厘米, 自然放置96小时, 取出, 置于滤纸上, 自然放置96小时后置于真空烘箱中, 真空加热150℃、干燥48小时以上, 移入手套箱中装配电池进行单电极的电池性能的测试。

典型的电极电池性能如下:

充放电电流密度:0.5mA/cm²

(1).在 1M LiClO₄ EC/DEC 电解液中

首次放电容量:279.5mAh/g, 充放电效率:76.8%

第二次放电容量:251.3mAh/g, 充放电效率:95.7%

第十次放电容量:248.0mAh/g, 充放电效率:97.3%

首次充放电效率提高 27.0%, 稳定循环容量提高 5.3mAh/g

(2).在 1M LiClO₄ PC/DME 电解液中

首次放电容量:217.5mAh/g, 充放电效率:63.8%

第二次放电容量:203.7mAh/g, 充放电效率:90.4%

第十次放电容量:198.1mAh/g, 充放电效率:94.9%

首次充放电效率提高 48.0%, 稳定循环容量提高 172.7mAh/g

实施例 5:

将MCMB10碳粉按实施例1中所述方法制备的粉末电极置于培养皿中, 加入5%的Li₂CO₃乙醇溶液, 使溶液液面超出电极表面2—3厘米, 自然放置96小时, 取出, 置于滤纸上, 自然放置96小时后置于真空烘箱中, 真空加热150℃、干燥48小时以上, 移入手套箱中装配电池进行单电极的电池性能的测试。

典型的电极电池性能如下:

充放电电流密度:0.5mA/cm²

(1).在 1M LiClO₄ EC/DEC 电解液中

首次放电容量:279.6mAh/g 充放电效率:73.5%

第二次放电容量:265.0mAh/g, 充放电效率:98.4%

第十次放电容量:255.3mAh/g, 充放电效率:98.3%

首次充放电效率提高 13.2%, 稳定循环容量提高 114.0mAh/g

(2).在 1M LiClO₄ PC/DME 电解液中

首次放电容量:249.0mAh/g, 充放电效率:60.2%

第二次放电容量:222.9mAh/g, 充放电效率:96.5%

第十次放电容量:203.2mAh/g, 充放电效率:98.9%

首次充放电效率提高 13.1%, 稳定循环容量提高 72.7mAh/g

实施例 6:

将MCMB10碳粉按实施例1中所述方法制备的粉末电极置于培养皿中, 加入5%的LiOH乙醇溶液, 使溶液液面超出电极表面2—3厘米, 自然放置96小时, 取出, 置于滤纸上, 自然放置96小时后置于真空烘箱中, 真空加热150℃、干燥48小时以上. 移入手套箱中装配电池进行单电极的电池性能的测试.

典型的电极电池性能如下:

充放电电流密度:0.5mA/cm²

(1).在 1M LiClO₄ EC/DEC 电解液中

首次放电容量:243.6mAh/g, 充放电效率:77.8%

第二次放电容量:223.5mAh/g, 充放电效率:94.3%

第十次放电容量:210.8mAh/g, 充放电效率:95.8%

首次充放电效率提高 17.5%, 稳定循环容量提高 69.5mAh/g

(2).在 1M LiClO₄ PC/DME 电解液中

首次放电容量:200.9mAh/g, 充放电效率:75.4%

第二次放电容量:216.7mAh/g, 充放电效率:89.3%

第十次放电容量:203.8mAh/g, 充放电效率:92.8%

首次充放电效率提高 28.3%, 稳定循环容量提高 73.3mAh/g

实施例 7:

将沥青碳纤维(T-3, 1200℃)碳粉按实施例1中所述方法制备的粉末电极置于培养皿中, 加入1%的Li₂O水溶液, 使溶液液面超出电极表面2—3厘米, 自然放置96小时, 取出, 置于滤纸上, 自然放置96小时后置于真空烘箱中, 真空

加热150℃、干燥48小时以上，移入手套箱中装配电池进行单电极的电池性能的测试。

典型的电极电池性能如下：

充放电电流密度： $0.5\text{mA}/\text{cm}^2$

(1).在 1M LiClO_4 EC/DEC 电解液中

首次放电容量： 346.5mAh/g ，充放电效率： 79.4%

第二次放电容量： 338.3mAh/g ，充放电效率： 92.1%

第十次放电容量： 327.5mAh/g ，充放电效率： 98.3%

首次充放电效率提高 32.1% ，稳定循环容量提高 44.6mAh/g

(2).在 1M LiClO_4 PC/DME 电解液中

首次放电容量： 297.4mAh/g ，充放电效率： 82.1%

第二次放电容量： 259.8mAh/g ，充放电效率： 94.9%

第十次放电容量： 241.3mAh/g ，充放电效率： 96.7%

首次充放电效率提高 36.9% ，稳定循环容量提高 23.3mAh/g

实施例 8：

将沥青碳纤维(T-3, 1200°C)碳粉按实施例1中所述方法制备的粉末电极置于培养皿中，加入10%的 Li_2O 水溶液，使溶液液面超出电极表面2—3厘米，自然放置96小时，取出，置于滤纸上，自然放置96小时后置于真空烘箱中，真空加热150℃、干燥48小时以上，移入手套箱中装配电池进行单电极的电池性能的测试。

典型的电极电池性能如下：

充放电电流密度： $0.5\text{mA}/\text{cm}^2$

(1).在 1M LiClO_4 EC/DEC 电解液中

首次放电容量： 318.6mAh/g ，充放电效率： 67.4%

第二次放电容量： 285.7mAh/g ，充放电效率： 92.4%

第十次放电容量： 279.3mAh/g ，充放电效率： 96.7%

首次充放电效率提高 20.1% ，稳定循环容量降低 3.6mAh/g

(2).在 1M LiClO_4 PC/DME 电解液中

首次放电容量： 292.5mAh/g ，充放电效率： 67.0%

第二次放电容量： 248.1mAh/g ，充放电效率： 90.5%

第十次放电容量： 234.3mAh/g ，充放电效率： 94.2%

首次充放电效率提高 21.8% ，稳定循环容量提高 16.3mAh/g



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